

इंटरनेट

मानक

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Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

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“Step Out From the Old to the New”

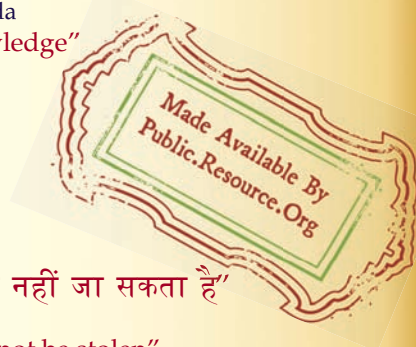
IS 13831 (1993): Pesticide - Method for Determination of Residues in Agricultural and Food Commodities, Soil and Water - Acephate and its Toxic Metabolite, Methamidofos [FAD 1: Pesticides and Pesticides Residue Analysis]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

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टाक्सिक मेटाबॉलाईट, मेथामिडोफॉस

Indian Standard

PESTICIDE — METHOD FOR DETERMINATION
OF RESIDUES IN AGRICULTURAL AND FOOD
COMMODITIES, SOIL AND WATER —
ACEPHATE AND ITS TOXIC METABOLITE,
METHAMIDOFOS

UDC 664 : 543 [632'95'028 ACE]

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NEW DELHI 110002

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Price Group 2

FOREWORD

This Indian Standard **was adopted by the Bureau of Indian Standards**, after the draft finalized by the Pesticides Residue **Analysis** Sectional Committee had been approved by the Food and Agriculture Division Council.

Acephate (0, **S-dimethyl** acetyl phosphoramidothioate) is used in agriculture for the control of insect pests. Assessment of its residues in food commodities is an important step in safeguarding **human** health.

This standard will enable the health authorities and others engaged in the field to follow uniform test **procedures** for the estimation of acephate and/or methamidofos residues in food commodities. In the preparation of this standard, due consideration has been given to the maximum limits of acephate **and/or** methamidofos residues laid under the provisions of ***Prevention of Food Adulteration Act, 1954*** and the Rules framed thereunder. The test method is restricted to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 :1960 'Rules for rounding off numerical values (***revised***).'

-PESTICIDE -METHOD FOR DETERMINATION OF RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES, SOIL AND WATER — ACEPHATE AND ITS TOXIC METABOLITE, METHAMIDOFOS

1

7.3 Methanol

Glass redistilled.

7.4 Acetonitrile

Glass redistilled.

7.5 Sodium Sulphate

Anhydrous.

7.6 Silica Gel for Column Chromatography

60-120 mesh.

7.7 Petroleum Ether

Boiling point 40-60°C.

7.8 Methanol in Ethyl Ether

5 percent (v/v).

7.9 Methyl Isobutyl Ketone

7.10 Methanol in Ethyl Ether

10 percent (v/v).

7.11 Acephate Reference Standard

Of known purity.

7.12 Metbamidofos Reference Standard

Of known purity.

7.13 Aqueous Sodium Chloride Solution

5 percent (m/v).

7.14 Methylene Chloride

AR grade.

8 EXTRACTION

8.1 Fruit and Vegetables

Transfer 50 g of finely chopped fruit or vegetable sample to a **waring** blender. Add 150 g anhydrous sodium sulphate, mix well add 150 ml ethyl acetate. Blend the mixture for 5 minutes. Filter the extract through a layer of anhydrous sodium sulphate on a filter paper mounted on the funnel. Transfer the residues on the filter paper back to the **waring** blender, add 100 ml ethyl acetate, blend for 5 minutes and filter again. Rinse the residue and the filter twice with 50 ml portion of ethyl acetate. Collect the filtrates and washings and evaporate the combined extracts to dryness in a vacuum rotary evaporator using water bath at 40-60°C.

8.2 Cereals and Grains

Transfer 50 g finely ground sample to a **waring** blender, add about 20 g anhydrous sodium sulphate followed by 150 ml ethyl acetate. Blend the mixture for 5 minutes and continue the extraction as described in 8.1.

8.3 Oilseeds and Nuts

Transfer 50 g of finely powdered sample to a **waring** blender, add about 50 g anhydrous sodium sulphate, mix well and add 150 ml ethyl acetate. Blend the mixture for 5 minutes and continue the extraction as prescribed in 8.1. After evaporation of the combined extract dissolve the residue in 50 ml acetonitrile and transfer the solution to a 125-ml separator-y funnel. Rinse the flask twice with 10 ml portion of acetonitrile adding the washing to the separatory funnel. Wash the acetonitrile solution thrice with 25 ml petroleum ether discarding the washings. Finally evaporate the acetonitrile solution to dryness in a vacuum, rotary evaporatory using water bath at 40-60°C.

8.4 Soil

Transfer 50 g of air dried and sieved soil into a 500-ml conical flask. Add about 200 ml of acetone. Shake the contents well on a rotary shaker for two hours. Filter extract through Buchner funnel with 20 ml additional acetone. Collect the extracts and evaporate off solvent on a vacuum rotary evaporator. Add about 100 ml of 5 percent aqueous sodium chloride solution (see 7.13) to a 250-ml separatory funnel. Dissolve the **residues** in 50 ml of methylene chloride and transfer the solution to the separatory funnel. Rinse the flask twice with 10 ml portions of methylene chloride, add the washings to the separatory funnel. Shake the contents in separatory funnel well for about 2 minutes. Allow the layers to separate. Drain off the lower methylene chloride layer through a layer of anhydrous sodium sulphate mounted on a funnel. Repeat the partitioning twice with 50 ml portions of methylene chloride. Wash the layer of sodium sulphate with 20 ml additional methylene chloride. Collect the methylene chloride extracts and finally evaporate to dryness in a vacuum rotary evaporator using water bath at 40-60°C.

8.5 Water

Transfer 300 ml of the water to a 250-ml separatory funnel. Add 50 ml of aqueous sodium chloride solution (see 7.13) followed by 75 ml of methylene chloride to the separatory funnel. Shake the contents well for about 2 minutes and allow the layer to separate. Drain off the lower organic layer through a layer of anhydrous sodium sulphate mounted on funnel. Repeat the extraction twice with 50 ml portions of methylene chloride. Wash the sodium sulphate layer with 10 ml additional methylene chloride. Collect the methylene chloride extracts and evaporate off to dryness in a vacuum rotary evaporator using water bath at 40-60°C.

9 CLEAN UP

9.1 Prepare a pre-washed silica gel **chromato-**graphic column by placing a glass wool in the

bottom of the column, add 50 ml ethyl ether and 15 g silica gel. Rinse the sides of the column with ether and place a 15 g layer of sodium sulphate over the silica gel. Again rinse the sides of the column with ether and allow the relevant to drain to the top of the column.

9.2 Using several 5 ml portions of ether, transfer the extracted residue (see 8.1-8.5) quantitatively or to the column. Wash the column with 100 ml ether followed by 100 ml -of 5 percent methanol in ether (see 7.8) discarding the eluate. Then elute acephate and methamidofos from the column with 200 ml 10 percent methanol in ether (see 7.10). Collect the eluate and evaporate to dryness in vacuum rotary evaporatory.

10 ESTIMATION

10.1 Preparation of Standard Solution

For reference standard, prepare solutions of acephate and methamidofos in methyl isobutyl ketone with concentrations of 0.01 to 10 $\mu\text{g/ml}$ and measure the peak area after each injection.

10.2 Preparation of Sample Solution

Dissolve the residue after clean up (see 9.2) in 2 ml methyl isobutyl ketone and inject 2 μl of this solution into the gas chromatograph. Identify the peaks for methamidofos and

acephate by their retention times and measure the peak area.

11 CALCULATION

Residue of captafol ($\mu\text{g/g}$)

$$= \frac{A_1 \times V_2 \times V_3 \times C}{A_2 \times V_1 \times M} \times f$$

where

A_1 = peak area of the sample;

V_2 = volume, in μl , of standard captafol injected;

V_3 = total volume, in ml, of the sample solution;

c = concentration, in $\mu\text{g/g}$, of the standard solutions;

f = recovery factor

$$= \frac{100}{\text{percent mean recovery}},$$

A_2 = peak area of the standard;

V_1 = volume, in μl , of the sample injected; and

M = mass, in g, of the sample taken for analysis.

NOTE — Percent mean recovery is determined by taking untreated control sample to which a known amount of acephate methamidofos is added and analyzed as described above.

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